



IN THE UNITED STATES PATENT & TRADEMARK OFFICE

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: GROUP ART UNIT:

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SIR:

4.1 Experiment

Tests on improving the shelf life of N-dimethylaminopropyl methacrylamide
(DMAPMA)

3 x 500 g of DMAPMA raw amide from industrial production, synthesized by titanium-catalyzed aminolysis of methyl methacrylate (MMA) with N-dimethylaminopropylamine, were outgassed in a Büchi evaporator at 65 °C and 2 mbar. The MMA-free raw amide was then distilled in quantities of 200 g each in the presence of added comparable quantities of stabilizers and stabilizer combinations at 130 °C and 1 mbar. The distillates, which were as pale as water and clear, were analyzed by gas chromatography (purity of > 99% in each case), and divided into halves, which were placed respectively into pale and brown 250-ml glass bottles and stored at room temperature in daylight. To test for polymer produced during storage, samples were taken at the times indicated in the table, diluted with 5 times their volume of n-hexane and evaluated visually for possible turbidity due to precipitated polymer.

4.2 Results

The results are summarized in the following Table.

Stabilizer	Bottle	0	1d	2d	6d	32d	73d	104d
None	pale	-	-	-	P			
	brown	-	-	-	-			
Phosphoric acid	pale	-	-	-	-	-	P	
	brown	-	-	-	-	-	-	
DEHA/nitroethane	pale	-	-	-	-	P		
	brown	-	-	-	-	-		
DEHA/cupferron	pale	-	-	-	-	-	-	-
	brown	-	-	-	-	-	-	-

Legend:

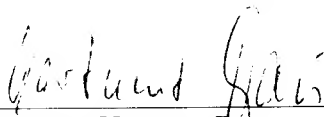
- no polymer formed
P onset of polymer formation
d day(s)
DEHA N,N-diethylhydroxylamine

cupferron (N-nitroso-N-phenylhydroxylamine ammonium salt)

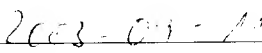
Only the DEHA/cupferron combination according to the present invention exhibited superior stabilization properties, so that no polymer was formed even after 104 days in either the pale or the brown bottle.

5. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

6. Further deponent saith not.



Signature, Hartmut Rau



Date